SHUYKIN, N.I.; KOMISSAROVA, N.L.

Synthesis and catalytic dehydrocyclization of 2- and 3-n-butylphenanthrenes. Izv.AN SSSR. Ser.khim. no.1:125-129 '66. (MIRA 19:1)

1. Institut organicheskoy khimii im. N.D.Zelinskogo AN SSSR. Submitted August 5, 1963.

KOMISSAROVA, L.N.; POKROVSKIY, B.I.; GRANOVSKIY, Yu.V.; SHAPLYGIN, I.S.

Solid solutions based on scandium oxide in the system $Sc_2O_3 - Fe_2O_3 - MnO_x$ studied by the statistical method of experiment planning. Zhur.neorg.khim. 11 no.1:151-155 Ja '66. (MIRA 19:1)

1. Moskovskiy gosudarstvennyy universitet imeni M.V.Lomonosova, kafedra neorganicheskoy khimii. Submitted March 27, 1965.

LIVANOVA, O.V., kand. tekhn. nauk (Moskva); KOMISSAROVA, I.P., inzh. (Moskva)

Experimental study of the heating of a solid rotor of a synchronous motor during asynchronous start. Elektrichestvo no.2:74-75 F '65. (MIRA 18:3)

NIKITIN, Viktor Vladimirovich; RUPASOV, Konstantin Andreyevich; KOMISSAROVA, I.S., red.; MAKAROVA, N.F., tekhn. red.

[Determinations of mathematical concepts in a secondary school course] Opredeleniia matematicheskikh poniatii v kurse srednei shkoly; posobie dlia uchitelei. Izd.2. Moskva, Uchpedgiz, 1963. 148 p. (MIRA 17:1)

KOMISSAROVA, K. V. (Gor'Kiy)

"Concerning the Algorithm of the Translation of English Radiotechnical Texts into Russian."

Theses - Conference on Machine Translations, 15 - 21 May 1958, Moscow.

1ST AND 2ND ORDERS

PROCESSES AND PROPERTIES INDEX

3RD AND 4TH ORDERS

CA

2

The equilibrium curve of styrene and ethylbenzene. I. I. Zeidler, L. A. Konstantsova and N. N. Golubitskaya. *J. Applied Chem.* (U.S.S.R.) 13, 1699-1702 (in French, 1702) (1940).—The equil. curve for the PhEt-PhCH:CH_2 mixt. was constructed at 50 and 90 mm. of residual pressure by the Othmer and Lea method (cf. *C. A.* 22, 3560). The equil. curves obtained were below the theoretical curves (a neg. variation from the Raoult law), and the curve at 90 mm. pressure was more convex than that at 50 mm. pressure. No polymerization was observed at temps. corresponding to the residual pressure of 90 mm. (under exptl. conditions). A. A. Podgorny

ASB-SLA METALLURGICAL LITERATURE CLASSIFICATION

KOMISSAROVA, L.A.

DOLGOV, B.N., professor; KOMISSAROVA, L.A.

Dehydrocyclization of heptene-3-OH-2. Nauch. biul. Len. un. no.22: 23-24 '49. (MLRA 10:4)

1. Kafedra organicheskoy khimii.
(Heptene)

KOMISSAROVA, L.A.

DOLGOV, B.N., professor; KOMISSAROVA, L.A.

Isomerization of halogen derivatives obtained through the interaction of magnesium organic compounds and dichloramine. Nauch. biul. Len. un. no.22:26 '49. (MLRA 10:4)

1. Kafedra organicheskoy khimii.
(Magnesium organic compounds)
(Chlorimids)

alcohol yields 30-35% ethyl acetate as the main reaction product; at 300-325°C, the main reaction product (60%) consists of acetone and higher ketones, such as methylpropyl ketone, methyl isobutyl ketone, dipropyl ketone, and methyl amyl ketone. A mechanism of formation of these ketones is proposed. Seven diagrams, 4 tables, 13 references (9 Russian: 1925-49)

Institution: None

Submitted : Jl 9, 1953

USSR/Chemistry - Lithium Aug 52

"New Data on the System Lithium Sulfate - Calcium Sulfate," V. Ye. Plyushchev and L. N. Komissarova, Moscow Inst of Fine Chem Technol imeni M. V. Lomo-nosov

"DAN SSSR" Vol 85, No 5, pp 1041-1043

Chemically pure Li_2SO_4 and $CaSO_4$ were used to obtain data for the diagrams. With 16.5 mole % of this compd at 852° a steadily climbs to 1205° at 78 mole %. From here on the curve has a different character due to oxide formation. Polymorphic conversion takes place at 575°. Submitted by Acad G. G Urazov 19 Jun 52.

239T15

239T15

KOMISSAROVA, L.N.

Investigation of interaction of lithium, sodium, and calcium sulfates in fusions. V. Ia. Pyatnitskiy, S. I. Zhdanov, L. N. Komissarova, and S. B. Dvadtseva. *Trudy Moskovsk. Inst. Tonkoy Khim. Tekhnol.*, 1953, No. 4, 10–18; *Referat. Zhur., Khim.* 1954, No. 33879.—The liquidus of the system Li_2SO_4-Na_2SO_4-$CaSO_4$ was studied by the method of thermal analysis. The highest melting temps. were in fusions adjacent to the apex of $CaSO_4$. The area of low melting temp. was adjacent to the system Li_2SO_4-Na_2SO_4 and extended far into the depth of the diagram. The lowest melting temp. was 544°. A study of internal sections of the ternary system and of the microstructure of solid phases indicated the formation of a continuous series of solid solns. in the area adjacent to the binary system Li_2SO_4-Na_2SO_4 and partly to the system Na_2SO_4-$CaSO_4$. Upon cooling the solid solns. undergo appreciable changes and decompn. Particularly was observed the crystn. of the double salt Li_2SO_4·Na_2SO_4. Changes of the solid solns. connected with polymorphism was also observed.

M. Hosch

KOMISSAROVA, L. N., kandidat khimicheskikh nauk, redaktor; L'VOVA, N.M., redaktor; BOGDANOV, V.P., tekhnicheskiy redaktor.

[Hafnium; collection of translations] Gafnii; sbornik perevodov. Moskva, Izd-vo inostrannoi lit-ry, 1955. 153 p. (MLRA 9:6)
(Hafnium)

KOMISSAROVA, L. N.

The reaction between sodium and calcium sulfates at high temperatures. [illegible] Komissarova, [illegible] and B. B. Stepina. Trudy [illegible] 1955, [illegible]

SO_4 — $CaSO_4$ was analyzed by thermal [illegible] methods at high temps., and a fusion diagram [illegible] are given. There is no compd [illegible] $4Na_2SO_4$ in the system, however Na_2SO_4 [illegible] was found in the solidus. A max [illegible] 20 moles % $CaSO_4$, characterizing [illegible] unstable compd. The eutectic [illegible] the eutectoid at 350° and [illegible]

Komissarova, L. N.

USSR/Thermodynamics. Thermochemistry. Equilibria. Physico-Chemical Analysis. Phase Transitions. B-8

Abs Jour : Ref Zhur - Khimiya, No 8, 1957, 26145

Author : V.Ye. Plyuchchev, L.N. Komissarova, L.V. Meshchaninova, L.M. Akulkina.

Title : Study of Interaction of Chlorides of Alkali and Alkali Earth Metals in Melts. III. Study of Interaction of Sodium, Potassium Calcium, Cesium, Rubidium and Lythium Chlorides in Melts.

Orig Pub : Zh. neorgan. khimii, 1956, 1, No 4, 820-833; corrections in No 12, 2874

Abstract : The ternary systems LiCl - Na Cl - $CaCl_2$(I), KCl - RbCl - $CaCl_2$ (II) and KCl - CsCl - $CaCl_2$ (III) were studied by the visual-polythermal method and the isoterms of the liquidus surfaces of the systems were plotted. The existence of two regions of primary crystallization of $CaCl_2$ and solid solutions of LiCl and NaCl was established in 1. It is shown that II is of the zonal type. The system has 3 crystallization fields: of $CaCl_2$, of a solid solution of KCl and RbCl,

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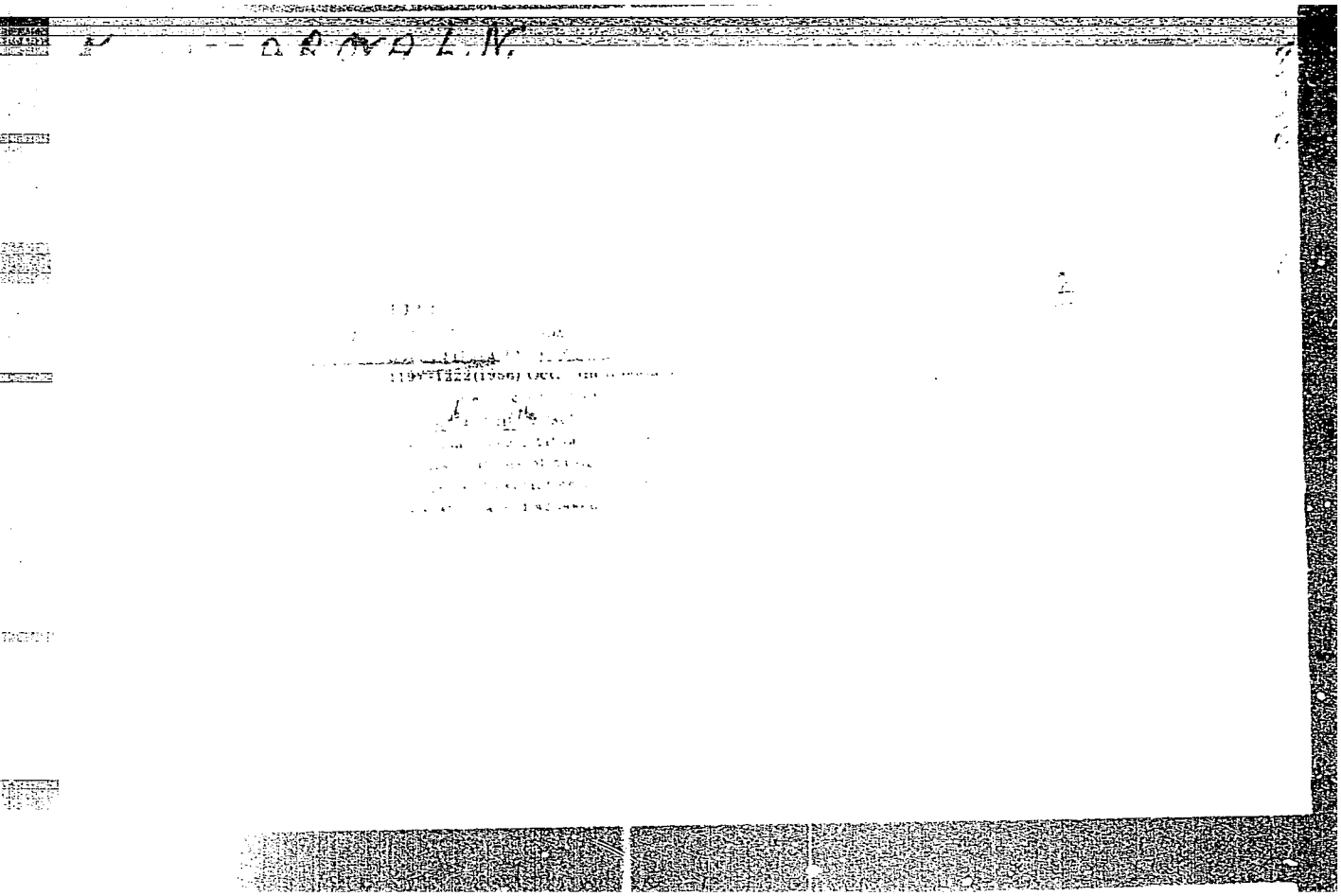
Inst. Fine Chem Technology im M.V. Lomonosov



Komissarova, L.N.

KOMISSAROVA, L.N., kand.khim.nauk, red.; PLYUSHCHEVA, V.Ye., kand.khim. nauk, red.; L'VOVA, N.M., red.; SHEMANINA, V.N., red.; SMIRNOVA, N.I., tekhn.red.

[Rare earth metals; a collection of articles. Translations.] Redkozemel'nye metally; sbornik statei. [Perevody.] Moskva, Izd-vo inostr.lit-ry, 1957. 419 p. (Redkie metally 57 La (138,9) - 71 Lu (175,0)) (MIRA 11:1)

(Rare earth metals)

153-58-1-6/29

AUTHORS: Komissarova, L. N., Plyushchev, V. Ye., Yuranova, L. I.

TITLE: An Investigation of the Thermal Stability of Zirconium-Sulfate-Tetrahydrate (Izucheniye termicheskoy ustoychivosti tetragidrata sul'fata tsirkoniya)

PERIODICAL: Izvestiya vysshikh uchebnykh zavedeniy. Khimiya i khimicheskaya tekhnologiya, 1958, Nr 1, pp. 37 -42 (USSR)

ABSTRACT: The above-mentioned sulfate-tetrahydrate is of great importance amongst the other zirconium-sulfates. Its method of production is given and the crystalline form is mentioned from publications (Reference 1). A survey of the publications on the problem referred to in the title, is given. Details on the condition of the material used for the tests and on the methods applied, are given in the experimental part. The results are summarized in tables 1 and 2. A thermogram covering the range between 20°C and 1100°C is given in figure 1. It shows 3 clear endothermic effects: the two first one between 130° and 215°C, which correspond to the separation of the water of crystallization; the 3rd effect (700 to 740°C) characterizes a complete decay of the sulfate with the separation of SO_3.

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(Figure 2). This figure shows the curve of the change of weight of the tetrahydrate which confirms and accurately defines the destructive character of this salt. The results of investigation of the dehydration- and decomposition-processes obtained by the methods described here, are compared in table 3.

Conclusions: 1) The last mentioned processes of dehydration and decomposition of zirconium-sulfate-tetrahydrate were investigated by means of a) Heating in air up to the attaining of a constant weight at various temperatures, b) Pyrometer by N. S. Kurnakov, and c) a continuous balance. 2) According to the velocity of heating, the dehydration of the tetrahydrate takes place either in 2 or 3 stages. In all cases, 3 water molecules within the range of 100 to 160°C are cracked at a time. One water molecule, on the other hand, is retained more vigorously and escapes slowly at graduate heating; at 190° to 215°C half of the quantity of the water gets lost up to the complete dehydration taking place at 300 to 340°C. 3) The decomposition of the zirconium sulfate is accompanied

An Investigation of the Thermal Stability of Zirconium-Sulfate-Tetrahydrate

by an escape of SO_3 and is gradually completed between 450 to 800°C. 4) The special solidity of the bond of water molecule points to the fact that the properties of tetrahydrate are more correctly expressed by the coordination-formula/$H_2ZrO(SO_4)_2$/.$3H_2O$. There are 2 figures, 3 tables, and 8 references, 6 of which are Soviet.

ASSOCIATION: Moskovskiyinstitut tonkoy khimicheskoy tekhnologii im. M. V. Lomonosova.Kafedra tekhnologii redkikh i rasseyannykh elementov (Moscow Institute for Fine Chemical Technology imeni M. V. Lomonosov, Professorial Chair for Rare and Dispersed Elements)

SUBMITTED: September 16, 1957

AUTHORS: Plyushchev, V. Ye., Shakhno, I. V., Komissarova, L. N., Nadezhdina, G. V. SOV/156-58-2-18/48

TITLE: Concerning Several Regularities in the Change of Solubility of the Alkali Metal Chlorides in Alcohols (O nekotorykh zakonomernostyakh izmeneniya rastvorimosti khloridov shchelochnykh metallov v spirtakh)

PERIODICAL: Nauchnyye doklady vysshey shkoly, Khimiya i khimicheskaya tekhnologiya, 1958, Nr 2, pp. 279 - 282 (USSR)

ABSTRACT: The problem referred to in the title was especially interesting from a practical point of view. There should be a way to separate the adjacent pairs of elements which always accompany one another (Li - Na, K - Rb, Rb - Cs). A literature search revealed that statements made about the solubilities of these alkali chlorides are widely contradictory. The theoretical aspects of the problem are interesting, but the practical are no less important, since single solvents can work specifically and selectively and make it possible by the solution of this particular problem to overcome other similar difficulties. On this basis the authors proceeded to carry out

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appropriate experiments at 0 - 70°. Solvents used were CH_3OH, C_2H_5OH, n.C_3H_7OH, n.C_4H_9OH, iso-C_4H_9OH(primary) and iso-$C_5H_{11}OH$ (primary). In the system with LiCl 5 - 6 days were allowed for the system to reach equilibrium. 6 - 7 days were allowed for the others. The solid phase, which was in equilibrium with the saturated solution was the original starting chloride. Distinct phases formed by the dissolution of LiCl in CH_3OH and in C_2H_5OH at 0°. They represented LiCl · $3CH_3OH$ and LiCl.$4C_2H_5OH$ (Ref 6). Table 1 shows the extreme solubility (in weight per cent) plus the range of temperature during the investigation. From this data the following peculiarities are emphasized: 1) The solubility of each chloride increases gradually with temperature. Only with the formation of the solvated form does the curve show a divergence, corresponding to the second branching. 2) This solubility increases with increasing molecular weight of both the normal and iso-alcohols. 3) LiCl is striking for its relatively high solubility in all alcohols. With the increasing atomic number the solubility of

Concerning Several Regularities in the Change of Solubility of the Alkali Metal Chlorides in Alcohols

SOV/156-58-2-18/48

the chloride changes rapidly, so that in the transition from LiCl to KCl it increases by 100 to 10 000 times, while it increases twelve-fold in the transitions from RbCl to CsCl. There are 1 table and 6 references, 2 of which are Soviet.

ASSOCIATION: Kafedra tekhnologii redkikh i rasseyannykh elementov Moskovskogo instituta tonkoy khimicheskoy tekhnologii im.M.V.Lomonosova (Chair of Technology of the Rare and Dispersed Elements of the Moscow Institute for Precision Chemical Technology imeni M.V. Lomonosov)

SUBMITTED: October 31, 1957

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5(2)
AUTHORS: Komissarova, L. N., Plyushchev, V. Ye. SOV/75-13-6-19/21

TITLE: Analytical Chemistry of Hafnium
(Analiticheskaya khimiya gafniya)

PERIODICAL: Zhurnal analiticheskoy khimii, 1958, Vol 13, Nr 6, pp 709-715
(USSR)

ABSTRACT: In both natural and industrial materials hafnium is always accompanied by zirconium. A survey of the numerous methods serving for the direct determination of zirconium, suitable for the hafnium determination as well, was recently given by Portcastle (Ref 2). To determine hafnium by these methods it is necessary to separate it from zirconium in the first place. For this purpose it is best to use ion exchange methods (Ref 1). The determination of hafnium in the presence of zirconium can be carried out by a few physical methods, as there are no such completely reliable and specific reagents in analytical chemistry to allow the determination of Hf or Zr when occurring together (Ref 3). On the other hand, highly specific reagents for the sum of both elements have been known for a long time. For the qualitative determination of hafnium in the presence

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of zirconium, exclusively optical and radiospectroscopic methods are used (Ref 4). Radiospectroscopic analysis is specially suitable for the quantitative determination (Refs 4-8). The quantitative determination of hafnium in the presence of zirconium can be carried out today by chemical, physico-chemical and physical methods. The chemical methods are all indirect. The present paper constitutes a very comprehensive synopsis of the literature concerning all these methods for the determination of hafnium in the presence of zirconium. There are 67 references, 14 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova i Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M. V. Lomonosova (Moscow State University imeni M. V. Lomonosov and Moscow Institute of Fine Chemical Technology imeni M. V. Lomonosov)

SUBMITTED: July 15, 1957

5 (2)

AUTHORS: Spitsyn, Vikt. I., Academician, Komissarova, L. N., Vladimirova, Z. A.

SOV/20-127-1-32/65

TITLE: Tungstates of Zirconium and Hafnium (Vol'framaty tsirkoniya i gafniya)

PERIODICAL: Doklady Akademii nauk SSSR, 1959, Vol 127, Nr 1, pp 120 - 123 (USSR)

ABSTRACT: The data given in publications on the substances mentioned in the title is very rare and contradicting (Refs 1-4). The present paper deals with the synthesis of hydrated and anhydrous tungstates and with the investigation of some of their properties. The first were obtained by the interaction between zirconyl- or hafnyl nitrate solutions and ammonium tungstate. Their molecular ratio was 1:1. Zr- or Hf hydroxide was precipitated when the pH of the solution amounted to more than 3.2. Colloidal precipitation was produced between pH 1.8 and 3.2 which coagulated in the case of heating in a NH_4NO_3 solution of 5%. Both initial substances reacted fully according to the analysis. Anhydrous tungstates were obtained by sintering (6 hours) oxides or hydroxides of the afore-mentioned elements with equimolar

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quantities of tungstic acid. The formation of the new phase was controlled by radiographic analysis. White fine-crystalline substances with radiographs which are very similar to one another are produced when the sintering products are chilled. The above tungstates are not produced if the chilling is carried out slowly. 1:1-compounds containing an excess of the component concerned were produced by sintering mixtures of ZrO_2 and HfO_2 with WO_3 in other ratios than 1:1, e.g. 1:2, 1:3, and 2:1. The radiographs did not show new lines indicating only 1:1 oxides. The compounds produced were analyzed by alkaline and pyrosulfate exposure. Table 1 shows the results. Accordingly, the substances synthesized are to be ascribed to the following formulas: $ZrOWO_4 \cdot 1.5H_2O$, $ZrOWO_4$, $HfOWO_4 \cdot 2H_2O$ and $HfOWO_r$. Hydrated zirconyl- and hafnyl tungstates are white radioamorphous substances which absorb humidity in air. Either the symmetry of the crystal lattices of anhydrous Zr- and Hf tungstates is low (their radiographs show more than 70 lines), or at least one of the axial parameters has high values. The high values of the angle of glide agree with the low density values: 5.27 for

$ZrOWO_4$, and 6.27 for $HfOWO_4$. The thermal stability, volatility with steam, and the behavior to the reagents of the afore-mentioned substances were investigated in order to confirm the individual character and to compare their properties. Figure 1 shows the curve of the change in weight, figures 2 and 3 the thermograms of heating. Dehydration is carried out in two stages and without a change of the amorphous state. Decomposition into the oxides ZrO_2, HfO_2 and WO_3 is caused by complete dehydration according to radiographic data. Volatility was checked according to reference 5 (Table 2). It is rather high in the two tungstates and increases with the content of bound water. Table 3 shows the behavior to HCl, H_2F_2, H_2SO_4, NaOH, and NH_4OH. There are 4 figures, 3 tables, and 5 references, 2 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova (Moscow State University imeni M. V. Lomonosov)

SUBMITTED: April 25, 1959

5 (2) SOV/20-128-1-24/58

AUTHORS: Men'kov, A. A., Komissarova, L. N., Simanov, Yu. P., Spitsyn, Viktor I., Academician

TITLE: On the Selenide and Telluride of Scandium

PERIODICAL: Doklady Akademii nauk SSSR, 1959, Vol 128, Nr 1, pp 92-94 (USSR)

ABSTRACT: The selenide and telluride of scandium were synthesized from elements by the authors. They are non-melting crystalline powders, the former of which is of brown-violet color and the latter black. The compounds obtained were investigated roentgenographically according to the powder method. Results of the analysis are given in tables 1 and 2. With the use of bromoform the density of selenide and telluride of scandium was determined pycnometrically at 22° (Table 3). The values of the density 4.52 g/cm³ (Ref 1) found for selenide of scandium are in good agreement with those from publications. The crystalline structure of selenide and telluride of scandium belongs to the type of structure $\gamma' = Al_2O_3$ (Ref 9). With respect to scandium ions the structures are defective. The lines Nr 6, 8, 11, 23 (Table 2) present with the telluride of scandium point to a partial transition of the $\gamma' = Al_2O_3$-structure to $\gamma = Al_2O_3$-

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structure with an ordered distribution of scandium ions. Similar structures are found with the telluride of indium In_2Te_3 (Ref 10) as well as with the selenide and telluride of gallium Ga_2Se_3, Ga_2Te_3 (Ref 11). These might be ascribed to the sphalerite type, however, with defects with respect to metal ions. The selenide and telluride of scandium must, however, be ascribed without doubt to the type $\gamma' = Al_2O_3$ on account of the presence of strong lines (200). There are 3 tables and 11 references, 3 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova (Moscow State University imeni M. V. Lomonosov)

SUBMITTED: June 11, 1959

89961
S/063/60/005/003/005/011/XX
A051/A029

5.2200 1043, 1273, 1228

AUTHORS: Yuranova, L.I., Komissarova, L.N., Plyushchev, V.Ye.

TITLE: On the Formation of Hexahydrates of Zirconium and Hafnium Oxynitrates

PERIODICAL: Zhurnal Vsesoyuznogo Khimicheskogo Obshchestva im. D.I. Mendeleyeva, 1960, Vol. 5, No. 3, p. 346

TEXT: The authors recently conducted a study on a new method for synthesizing $ZrO(NO_3)_2 \cdot 2H_2O$ and a similar compound of Hafnium, i.e., the dihydrates of zirconium and hafnium oxynitrates. It is possible to obtain individual zirconium and hafnium oxynitrates free of admixtures of any other nitrates. The results could be successfully reproduced several times. Numerous experiments showed that both substances crystallize in a wide range of HNO_3 concentrations and of the zirconium and hafnium concentration, as well as under various temperature conditions. While studying the system

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On the Formation of Hexahydrates of Zirconium and Hafnium Oxynitrates

difficult to crush and which resembled magnesium cement. In the second case mixtures were obtained in the form of a gruel, which after a certain time hardened into a non-transperent, white, enamel-like mass. The obtained preparations were thoroughly ground, dried in air to a constant weight and were analyzed for MeO_2, N_2O_5 and H_2O content. The MeO_2 content was determined by calcinating the corresponding hydrates to MeO_2 at 900°C. The Devard method was used for determining N_2O_5. The amount of water was calculated from the difference. The results of the analysis are given in the table and represent the average values of 5 determinations. It is pointed out that in repeated experiments the results were systematically and favorably reproduced. Thus, the composition of the obtained compounds is expressed by: $ZrO(NO_3)_2 \cdot 6H_2O$ and $HfO(NO_3)_2 \cdot 6H_2O$. X-ray findings were also obtained, which confirmed the formation of new phases. The hexahydrates were found to be well soluble in water and stable in air. Their densities at 20°C were estimated pycnographically and found to be 2.08 ± 0.02 and 2.66 ± 0.02, respectively. There is 1 table and 3 non-Soviet references.

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89961

S/053/60/005/003/005/011/XX
A051/A029

On the Formation of Hexahydrates of Zirconium and Hafnium Oxynitrates

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M.V. Lomonosova (Moscow Institute of Fine Technology, im. M.V. Lomonosov)

SUBMITTED: December 29, 1959

Table:

Preparation	Content, weight %			Molar ratio
	MeO_2	N_2O_5	H_2O	$MeO_2:N_2O_5:H_2O$
zirconium nitrate	36.30	31.85	31.85	1.0:1.02:6.1
hafnium nitrate	50.00	25.11	24.89	1.0:0.98:5.9

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5.2640
5.2200

69051

AUTHORS: Komissarova, L. N., Plyushchev, V. Ye., Kremenskaya, I. N. S/078/60/005/03/014/048 B004/B002

TITLE: Investigation as to Solubility and Thermal Stability of Zirconium Oxychloride

PERIODICAL: Zhurnal neorganicheskoy khimii, 1960, Vol 5, Nr 3, pp 586-592 (USSR)

ABSTRACT: First, the authors give a survey of publications on $ZrOCl_2 \cdot 8H_2O$, and mention L. K. Akhrap-Simonova (Ref 15). According to it, there are discrepancies as to solubility and stability of this compound, which urged the authors to write the present paper. The investigation was carried out with spectrally pure $ZrOCl_2 \cdot 8H_2O$. The removal of HfO_2 was achieved by means of ion exchange chromatography. The solubility was investigated in the Wobser ultrathermostat type U-8 (Table 1, Fig 1) within the range -2 to +110°. It was found that the solubility of $ZrOCl_2 \cdot 8H_2O$ increases with the temperature being raised to 70.5°. The maximum is reached at 42.00 weight% of ZrO_2. Below 70.5° no hydrolysis was observed in the concentrated solutions, as was observed by I. V. Tananayev and L. S. Guzeyeva (Ref 21) in diluted solutions. Above 70.5° the solubility of zirconium oxychloride is reduced due to hydrolysis and the development of compounds with a low content of chlorine of the general composition $mZrOCl_2 \cdot nZrO(OH)Cl \cdot pH_2O$ ($m > n$, $p < 8$). At 110°, a homogeneous, viscous

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KOMISSAROVA, L. N.

81939
S/078/60/005/07/04/014
B004/B056

15.2210

AUTHORS: Komissarova, L. N., Simanov, Yu. P., Vladimirova, Z. A.

TITLE: Some Properties of the Crystalline Modifications of ZrO_2

PERIODICAL: Zhurnal neorganicheskoy khimii, 1960, Vol. 5, No. 7, pp. 1413-1417

TEXT: In the introduction the authors discuss published data on the modifications of zirconium dioxide (Refs. 1-20). They then give a report on their investigations of the phase transformations of ZrO_2 within the temperature range of 20-1300°C and the reactivity of the various modifications. $Zr(OH)_4$ was produced from $Zr(SO_4)_2 \cdot 4H_2O$ by precipitation with ammonia. Thermal analysis was carried out by means of the Kurnakov pyrometer of the type ФПК-55 (FPK-55), the gravimetrical analysis by means of the continuous scales of the type ВР-НВ-20 (VR-NV-20). The X-ray pictures were taken by means of a БСВ (BSV)-tube and an РКД-57 (RKD-57)-camera. Fig. 1 shows the changes in the weight of $Zr(OH)_4$

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Some Properties of the Crystalline Modifications of ZrO_2

31939
S/078/60/005/07/04/014
B004/B056

during heating, Fig. 2 the thermogram, and Table 1 the radiographical data. The reactivity of the modifications of ZrO_2 was investigated by treatment with HCl and H_2SO_4 of various concentrations (Table 2). The results are: The tetragonal modification of ZrO_2 crystallizes within the temperature range of from 290 to 300°C with the thermal decomposition of zirconium hydroxide and -nitrate, and in the temperature range of from 350 to 400°C with the thermal decomposition of zirconium oxychloride. A further rise of temperature leads to the formation of the monoclinic modification. From the Debye patterns, the parameters a = 5.08 kX; c = 5.16_8 kX were obtained for the tetragonal modification; the parameters a = 5.11_7 kX; b = 5.19_2 kX; c = 5.29_9 kX; β = 80.82° were determined for the monoclinic modification. The reaction with HCl and H_2SO_4 showed that the tetragonal modification of ZrO_2 is considerably more reactive than the monoclinic one. Up to the range of the reversible transformation at 1170-1200°C, the reactivity of monoclinic ZrO_2 is independent of

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annealing temperature. However, samples which were heated beyond this temperature and were subjected to the transformation, showed a considerably lower degree of reactivity. There are 2 figures, 2 tables, and 20 references: 2 Soviet, 1 British, 1 Dutch, 6 German, 1 Italian, and 9 American.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova Kafedra neorganicheskoy khimii (Moscow State University imeni M. V. Lomonosov, Chair of Inorganic Chemistry)

SUBMITTED: March 10, 1959

KOROVIN, S.S.; GRIBENIK, Ye.N.; KOMISSAROVA, L.N.

Extraction of hafnium with tributyl phosphate. Zhur. neorg. khim. 5 no.8:1876-1881 Ag '60. (MIRA 13:9)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M.V. Lomonosova, Kafedra tekhnologii redkikh i rasseyannykh elementov.

(Butyl phosphate) (Hafnium--Analysis)

S/078/60/005/010/024/030/XX
B017/B067

AUTHORS: Spitsyn, Vikt. I., Komissarova, L. N., Shatskiy, V. M., and Pushkina, G. Ya.

TITLE: Study of the Complex Ammonium Scandium Carbonate

PERIODICAL: Zhurnal neorganicheskoy khimii, 1960, Vol. 5, No. 10, pp. 2223-2228

TEXT: The authors determined the optimum conditions for producing ammonium scandium carbonates, and described the properties of these compounds. The compound $NH_4Sc(CO_3)_2 \cdot 1.5H_2O$ was produced by dissolving freshly produced scandium hydroxide in a concentrated solution of ammonium carbonate, and subsequent crystallization at room temperature. This compound is stable at room temperature, and decomposes only at 95°C under formation of difficultly soluble basic scandium carbonate whose composition is not constant. The thermal decomposition of ammonium scandium carbonate was thermographically studied by means of a Kurnakov pyrometer. It was observed that the ammonium scandium carbonate decomposes gradually. At 140-190°C,

Card 1/3 [illegible] and 4 non-Soviet

Study of the Complex Ammonium Scandium Carbonate

S/078/60/005/010/024/030/XX
B017/B067

references.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova, Kafedra neorganicheskoy khimii (Moscow State University imeni M. V. Lomonosov, Chair of Inorganic Chemistry)

SUBMITTED: July 8, 1959

Card 3/3

S.2200

69510

S/020/60/131/04/039/073

B011/B017

AUTHORS: Spitsyn, Vikt. I., Academician, Komissarova, L. N., Vladimirova, Z. A., Simanov, Yu. P., Tyutyuyeva, N. N.

TITLE: Niobate and Tantalate of Zirconium

PERIODICAL: Doklady Akademii nauk SSSR, 1960, Vol 131, Nr 4, pp 857-860 (USSR)

TEXT: The authors describe the conditions of formation of zirconium tantalate and -niobate. Mixtures of zirconium- and niobium hydroxide (ZrO_2:Nb_2O_5 = 2:1, 1:1 and 1:2) served for their production. Besides these mixtures, also the individual hydroxides were sintered and/or roasted in silite furnaces at 1300°. Figure 1 shows the X-ray photographs which were taken on an iron anode with a camera of type RKD-57. They were measured by means of a comparator. The results are in good agreement with data from publications. The lines characteristic of ZrO_2 and Nb_2O_5 do not appear on the X-ray photograph with an oxide ratio of 2:1. Hence, a new phase was formed (Fig 1). No lines with a different oxide ratio than that mentioned were observed. Zirconium tantalate was produced by a similar method from the corresponding hydroxides (ZrO_2:Ta_2O_5 = 2:1) by sintering. The X-ray photograph showed no lines of ZrO_2, only some lines which might be ascribed to

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Niobate and Tantalate of Zirconium

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are highly resistant to HCl (36%), H_2F_2 (25%), H_2SO_4 (94%), and NaOH (40%). They were best dissolved in H_2F_2 where tantalate is more resistant. It is practically insoluble in hot-concentrated HCl- and H_2SO_4 solutions, in H_2SO_4 and ammonium sulfate mixtures. Also together with sodium pyrosulfate, K_2CO_3, and sodium peroxide it cannot be melted. The undissolved portion of the two zirconyl salts remains unchanged which indicates a high chemical resistance of these compounds. There are 2 figures, 2 tables, and 5 references.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova (Moscow State University imeni M. V. Lomonosov)

SUBMITTED: December 22, 1959

S/020/60/135/002/033/036
B016/B052

AUTHORS: Borisenko, L. F. and Komissarova, L. N.

TITLE: Scandium in the Minerals of the Tungstite Group

PERIODICAL: Doklady Akademii nauk SSSR, 1960, Vol. 135, No. 2, pp. 430-433

TEXT: The authors discuss the distribution character of scandium in three minerals of the tungstite group: hübnerite, tungstite, and ferberite. Against all expectations (V. M. Gol'dshmidt, Ref. 2; Ref. 3), there exists no direct relation between the content of Sc and that of divalent iron. The author proved an increased Sc content in all of the three above metals, irrespective of the ratio FeO : MnO. The material collected and evaluated by the authors shows that the average Sc_2O_3 content in the minerals of the tungstite group of greisen-type deposits exceeds that of typically hydrothermal deposits by at least one order of magnitude (Ref. 5). In the former case, scandium-bearing tungstite is in paragenesis with high-temperature quartz, topaz, mica, beryl, and some

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Scandium in the Minerals of the Tungstite Group

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B016/B052

other minerals (Table 2). In the latter case, however, paragenesis occurs mainly between Sc-bearing tungstite, hübnerite, and sulfides: pyrrhotite, pyrite, chalcopyrite, arsenopyrite, and bismuthinite. The authors explain the occurrence of the whole series of Sc-bearing minerals in the tungstite group among minerals of high-temperature paragenesis by the small difference in the lattice energies (U in kcal/mole) of their outermost links. Among the above compounds, however, Sc-bearing tungstite and ferberite occur more frequently than Sc-bearing hübnerite since the lattice energy of the two first-mentioned minerals is higher. In various deposits, the formation of Sc-bearing ferberite, tungstite, or hübnerite was dependent on the composition of the solutions. When examining tungstites of different genesis, the authors found that the physico-chemical conditions in the neighborhood of the forming ore body, namely, temperature, pressure and kind of solution determined the composition of the Sc-bearing minerals and their Sc content. The higher the temperature and pressure during the sedimentation of the minerals, the faster was the inclusion of scandium in the minerals of the tungstite group. If these minerals occur among those of high-temperature

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Scandium in the Minerals of the Tungstite Group

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paragenesis, they often contain larger amounts of Sc. The Sc content in minerals of the tungstite group belonging to the lower-temperature formation, is usually low or below the sensitiveness of the analytical method applied. The authors believe that scandium was included in the lattices of these minerals as scandium tungstate, niobate, or tantalate. They synthesized such highly stable compounds. Analyses by P. V. Diomidova, I. A. Bogdanovich, V. I. Tsirel'nikov, M. V. Kukharchik, V. M. Senderova, S. N. Fedorchuk, M. M. Povilaytis, and N. V. Lizunov, and a paper by A. Ye. Fersman (Ref. 6) are mentioned. L. I. Agapova supplied mineral samples. V. M. Shatskiy cooperated in the synthesis of tungstates. There are 2 tables and 6 references: 4 Soviet and 1 German.

ASSOCIATION: Institut mineralogii, geokhimii i kristallokhimii redkikh elementov Akademii nauk SSSR (Institute of Mineralogy, Geochemistry, and Crystallochemistry of Rare Elements of the Academy of Sciences USSR). Moskovskiy gosudarstvennyy Universitet im. M. V. Lomonosova (Moscow State University imeni M. V. Lomonosov)

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Scandium in the Minerals of the Tungstite Group

S/020/60/135/002/033/036
B016/B052

PRESENTED: April 14, 1960, by D. I. Shcherbakov, Academician

SUBMITTED: April 13, 1960

Card 4/4

KOMISSAROVA, L.N., kand. khim. nauk, red.; PLYUSHCHEV, V.Ye., kand. khim. nauk, red.; L'VOVA, I., red.; KHAR'KOVSKAYA, L., tekhn. red.

[Methods for the separation of rare earth metals; collection of translated articles] Metody razdeleniia redkozemel'nykh metallov; sbornik statei. Moskva, IZd-vo inostr. lit-ry, 1961. 361 p. (MIRA 15:2)

(Rare earth metals)

S/020/61/136/002/020/034
B016/B060

AUTHORS: Komissarova, L. N., Yuranova, L. I., and Plyushchev, V. Ye.

TITLE: Synthesis and Thermal Stability of Dihydrates of Oxy-nitrates of Zirconium and Hafnium

PERIODICAL: Doklady Akademii nauk SSSR, 1961, Vol. 136, No. 2, pp. 350-353

TEXT: A study has been made of the synthesis of zirconium and hafnium oxy-nitrate dihydrates and their thermal stability. While data available in the literature on the former dihydrate are insufficient and contradictory, the latter is as yet undescribed. Zirconium (hafnium-) oxychloride octohydrate, which contains a constant amount of crystal water, has proved to be the only usable initial substance for the synthesis of these compounds. A weighed-in portion of these salts was treated with 100% HNO_3 (MeO_2 : HNO_2 = 1 : 3 to 1 : 6). 1 : 4.5 was found as the optimum ratio. Air was blown through the solutions until the yellow color disappeared, and at 60°C they were vaporized. On the basis of the analysis,

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Synthesis and Thermal Stability of Dihydrates of Oxy-nitrates of Zirconium and Hafnium

S/020/61/136/002/020/034
B016/B060

the following formulas are ascribed to the compounds synthesized: $ZrO(NO_3)_2 \cdot 2H_2O$ and $HfO(NO_3)_2 \cdot 2H_2O$. They are white crystalline substances. The data obtained for equilibrium displayed certain differences between the zirconium and hafnium compounds regarding the stability and decomposition on heat treatment. They are, however, both thermally unstable and decompose completely at 400°C, when monoclinic ZrO_2 results from zirconium oxy-nitrate dihydrate with a 54.1% loss of weight. The hafnium compound displays a greater thermal stability, especially the monohydrate forming in between. Both substances decompose stepwise (the stages are less marked in the zirconium compound). There are 2 figures, 2 tables, and 9 non-Soviet references: 3 French, 1 British, and 4 German.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M. V. Lomonosova (Moscow Institute of Fine Chemical Technology imeni M. V. Lomonosov)

PRESENTED: June 24, 1960, by V. I. Spitsyn, Academician

SUBMITTED: June 22, 1960

Card 2/2

25860
S/020/61/139/004/020/025
B103/B220

18.3100

AUTHORS: Spitsyn, Vikt. I., Academician, Komissarova, L. N., and Men'kov, A. A.

TITLE: Production and properties of metallic scandium

PERIODICAL: Akademiya nauk SSSR. Doklady, v. 139, no. 4, 1961, 903-

TEXT: The production of metallic scandium from its anhydrous chloride $ScCl_3$ was studied, and its still little known properties were analyzed. The relatively high melting point of 1539°C, low specific gravity, considerable mechanical strength, and (under certain conditions) low chemical activity render scandium a promising material in several fields of modern technology. Spectroscopically pure scandium oxide obtained by thiocyanate extraction and precipitation of scandium oxalate from commercial Sc_2O_3 was used as initial substance to produce Sc. Anhydrous $ScCl_3$ was obtained by chlorination of the mixture scandium oxide + charcoal from sugar (3:1) in a quartz tube at 1000°C, and sublimed. It was reduced with metallic

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Production and properties of metallic ...

calcium in pure argon at 900°C. The reaction mixture containing about 5% Ca was filled into a tantalum crucible. The reaction products contained Ca, CaO, $ScCl_3$, and Si. They were pulverized (grain size <0.2 mm), treated with water, with 10% NaOH, again with water, and finally with methanol and ether. The solvent was decanted. The powdery metallic scandium obtained was dried in air (10-15min) and in vacuo (10^{-4}mm Hg, ~30min). Then, scandium was melted at reduced argon pressure (200 mm Hg) in an arc furnace. Previously, the metal had been pressed into tablets under a pressure of 100 kg/cm^2, and heated in high vacuum (10^{-5} - 10^{-6} mm Hg). The molten metallic scandium is a silvery metal with a characteristic yellow glimmer. It contains 97 - 97.5% Sc (analysis by the hydrogen method), whereas the gravimetric and volumetric methods gave corresponding values of 98 - 99% by weight. Small quantities of Si (0.1% by weight) and Ca (0.001%) were spectroscopically identified in most specimens. The analysis of molten Sc yielded in %: Sc 98 - 99; Cl<0.05; Ca<0.001; Si 0.1; O_2<0.9. Zr, Th, Y, Yb, Fe in total <0.1. For further purification molten Sc was sublimed in high vacuum from a tantalum crucible to a

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tantalum plate at 1500-1600°C by using a high-frequency apparatus with tube generator. The Sc content in the sublimate was < 99%. Radiographs of Sc sublimed in vacuo were analyzed. All 29 lines of the picture were easily indicated in an Mg-type hexagonal lattice with a = 3.302±0.005 kX and c = 5.255±0.005 kX, c/a = 1.591; Z = 2. The radiographic density is 2.992 g/cm^3, the pycnometric density = 3.0 g/cm^3. The cubic phase described in Ref. 13 (K. Meisel, Naturwiss., 27, 230 (1939)) and Ref. 6 (J. C. Achard et al., C. R., 243, 493 (1956)) is explained as being due to considerable impurities, mainly ScN, in the Sc metal. The device with diamond pyramid was used for determining the microhardness. For Sc > 99%, it was 75 ± 5 kg/mm^2, whereas 145 ± 10 kg/mm^2 was measured for Sc 97 - 97.5%. Further data refer to Sc 97 - 97.5%. The yield strength was determined on turned specimens of 2 mm diameter by means of a tensile-testing machine. The yield strength decreases considerably with increasing content of non-metallic additions. The stability in air was tested (a) isothermally at 20°C, and (b) polythermally between 20 and 800°C. Ad (a): A damped quartz balance was used (with assistance of V. A.

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Production and properties of metallic ...

Arslambekov, Institut fizicheskoy khimii AN SSSR, Institute of Physical Chemistry AS USSR). The tests showed that the metal surface was coated by an oxide film ~600 Å thick, whereupon oxidation stopped. Ad (b): Oxidation in air was studied by using a continuous balance. Metal powder (<0.2 mm) begins to oxidize at 250°C. The kinetics of interaction with aqueous HCl solutions was recorded on Sc plates (apparent surface 3-4 cm^2, weight 0.3 - 0.5 g) based on the rate of H_2 separation, and checked by the decrease in weight of Sc. The two methods gave corresponding data. Interaction between metallic Sc and HCl solutions occurs rather rapidly at HCl concentrations between 0.05 and 0.1 N and more. If the concentration of HCl is reduced, the dissolution of metal is rapidly showed down. In 0.001 N HCl (pH 3), the dissolution constant K is very low ($<5.10^{-5}$ $mg/cm^2 \cdot min$). Consequently, the authors state that practically no further dissolution of Sc takes place at this concentration, and the more so in H_2O. Yu. P. Simanov is thanked for discussing data obtained by X-ray analysis. There are 4 figures, 1 table, and 16 references: 6 Soviet-bloc

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and 10 non-Soviet-bloc. The most important references to English-language publications read as follows: Ref. 7: Chem. Age 82, 2106, 742 (1959); Ref. 9: F. H. Spedding & al. Trans Metallurg. Soc. AIME 218, No. 4, 608 (1960).

ASSOCIATION: Moskovskiy gosudarstvenny universitet im. M. V. Lomonosova (Moscow State University imeni M. V. Lomonosov)

SUBMITTED: April 15, 1961

X

28652

S/020/61/139/006/019/022
B103/B101

5.2200

AUTHORS: Tsirel'nikov, V. I., Komissarova, L. N., and Vikt. I. Spitsyn, Academician

TITLE: Thermal conductivity and viscosity of zirconium- and hafnium tetrachloride vapors in the temperature range 300-700°C

PERIODICAL: Akademiya nauk SSSR. Doklady, v. 139, no. 6, 1961, 1389-1391

TEXT: The authors determined: 1) thermal conductivity, 2) viscosity of the vapors of a) zirconium-, b) hafnium tetrachloride between 300 and 700°C. The above data are required for the development of new methods of separating a) and b). Ad 1): The apparatus used was a double furnace with dural blocks whose temperatures were measured by an automatic electronic potentiometer type ЭПП-09 (EPP-09). For measuring the thermal conductivity a platinum filament (thickness: 0.05 mm) stretched along the longitudinal axis of a pyrex tubule, was used. A platinum resistance thermometer indicating the wall temperature of the tubule, is bifilarly coiled upon it. The platinum filament is electrically heated in the atmosphere of the gas to be examined. At constant amperage, the

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resistance depends on the temperature of the filament and thus on the thermal conductivity of the gas. The temperature is measured in a resistance bridge. First, the resistances of platinum filament and thermometer are measured at room temperature, and then diagrams of the temperature dependence of their resistances are plotted. After placing the tubule into the furnace, the temperature difference between platinum filament and tubule walls as depending on the amperage of the current heating the platinum filament are measured at various wall temperatures, either in vacuo (10^{-3} mm Hg), or in dry hydrogen. Then, the tubule is filled with powdery (a) or (b), is evacuated to 10^{-3} mm Hg, sealed, and again put into the furnace. The lower block is heated up to 300-320°C. At this temperature, the vapor pressures of (a) or (b) are approximately 0.75 atm; the thermal conductivity of the vapor is independent of the temperature. The temperature of the upper block is varied between 350 and 500°C. Method of measurement: at a given wall temperature between 350 and 500°C, the temperature of the platinum filament and the current consumption are measured. These data serve for setting up the diagrams of the dependence of W on ΔT at given wall temperatures, and that of W on the wall temperature at constant $\Delta T = 20°C$. (1) is calculated according to the

equation $\lambda = \frac{\lambda_1 (W_2 - W_0)}{W_1 - W_0}$ where λ_1 is the thermal conductivity of hydrogen at the temperature concerned, W_0, W_1, and W_2 are the amperages required for generation of a temperature difference of 20°C in vacuo, in H_2, and in the vapors of (a) and (b), respectively. As expected, the coefficients of the thermal conductivities of (a) and (b) vapors increase with increasing temperature. This function is linear. The thermal conductivity of (a) is higher than that of (b). This difference increases with increasing temperature. At 300°C, the coefficients of the thermal conductivity are $4.31 \cdot 10^5$ and $3.67 \cdot 10^5$; at 500°C they are $6.35 \cdot 10^5$ and $4.89 \cdot 10^5$ cal/cm·sec·deg. Ad 2). The authors applied the method of vapor discharge through a capillary (Ref. 4, see below). The viscosity coefficient was calculated from the Hagen-Poiseuille equation. The values required for this: vapor pressure at inlet and outlet of the capillary; the quantity of vapor passing the capillary within a certain time, and the capillary parameters, were experimentally determined. The furnace used had a nickel and a dural block. The temperature of the nickel block was measured by an automatic

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potentiometer type ЭПВ-2 (EPV-2) between 350 and 700°C, that of the dural block by a chromel-alumel thermocouple with a ПП (PP) potentiometer, and was controlled by a special system (between 250 and 350°C). The quartz phial with a capillary fused on to it, was filled with freshly sublimed (a) or (b). Method of measurement: After filling up to approximately 1/3, the phial is weighed and placed into the furnace which is heated up to a given temperature. The phial is placed into the dural block and the capillary into the nickel block. After 5-10 min, the phial is taken out, cooled down and weighed. Thus, the amounts of (a) or (b) which passed the capillary, are measured. The vapor pressures of (a) and (b) are calculated according to the equations $\log P_{mm} = -5400/T + 11.766$ for (a), and $\log P_{mm} = -5197/T + 11.712$ for (b). The temperature of the dural block is chosen such as to guarantee a pressure of 200 mm Hg in the phial. The viscosity coefficient of the vapor at a given temperature of the capillary is calculated from the formula $\eta = \frac{\pi \cdot r^4 \cdot \tau \cdot \rho}{8ML} \frac{p_1^2 - p_2^2}{2p_1}$ containing a correction for the gas expansion in the capillary, with r denoting the

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radius of the capillary, p_1 being the pressure in the phial, and p_2 being the atmospheric pressure, τ denoting the time of the experiment, M being the weight loss of the phial, L being the length of the capillary, and ϱ being the vapor density in the capillary at given pressure and temperature. ϱ is calculated on the basis of the gas laws by considering the vapor to be an ideal gas. The measurements were conducted between 350 and 700°C. At these temperatures, the viscosity coefficients were directly proportional to the temperature. The viscosity of (b) is somewhat higher than that of (a). With raising temperature it increases somewhat more than that of (a). It was found at 500°C:

$\eta_{ZrCl_4} = 2640 \cdot 10^{-7}$ poise; $\eta_{HfCl_4} = 3505 \cdot 10^{-7}$ poise. There are 4 figures, 2 tables, and 5 references: 1 Soviet and 4 non-Soviet. The reference to the English-language publication reads as follows: Ref. 4: A. O. Rankin, Proc. Roy, Soc., A88, 575 (1918).

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova (Moscow State University imeni M. V. Lomonosov)

28652
S/020/61/139/006/019/022
B103/B101

Thermal conductivity and viscosity of...

SUBMITTED: April 28, 1961

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29018

S/020/61/140/004/019/023
B106/B110

5.2100

AUTHORS: Yuranova, L. I., Komissarova, L. N., and Plyushchev, V. Ye.

TITLE: New data on the behavior of zirconium and hafnium oxynitrates in aqueous solutions and organic solvents

PERIODICAL: Akademiya nauk SSSR. Doklady, v. 140, no. 4, 1961, 855-858

TEXT: Almost all studies described in the literature with regard to the chemistry of zirconium nitrate solutions concerned microquantities of zirconium and highly diluted solutions (Ref. 3: V. I. Paramonova, ZhNKh, 1, 1905 (1956); Refs. 4-6, see below). In practice, the behavior of zirconium and hafnium oxynitrates in more concentrated solutions is interesting, particularly when studying extraction methods for separating zirconium from hafnium. Therefore, the authors investigated the dependence of pH value, specific electrical conductivity, density, and viscosity of zirconium and hafnium oxynitrate solutions on the concentrations of these compound in aqueous solutions. Moreover, the solubility of oxynitrates in organic solvents was studied. The compounds $ZrO(NO_3)_2 \cdot 2H_2O$, $ZrO(NO_3)_2 \cdot 6H_2O$, and $HfO(NO_3)_2 \cdot 2H_2O$, $HfO(NO_3)_2 \cdot 6H_2O$ were

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tested. Figs. 1 and 2 show results obtained for pH value and specific electrical conductivity of aqueous solutions of these compounds. Since the hydrolysis of oxynitrates decreases with increasing concentration, the decrease of the pH value is only due to the increasing absolute quantity of dissolved salts. Hydrolysis of zirconium and hafnium oxynitrates in aqueous solutions was found to depend on time. The state of equilibrium is attained only two weeks after the solutions have been prepared. A rise in temperature effects stronger hydrolysis. As expected, oxynitrates of hafnium proved to be stronger bases than those of zirconium. The density and viscosity values measured for aqueous solutions of the oxynitrates concerned are shown in Figs. 3 and 4. 15 organic compounds of different classes were selected to study the solubility of zirconium and hafnium oxynitrates in organic solvents. Measurements were made at 20 and 30°C; temperature fluctuations were $\pm$ 0.1°. In all cases, solution equilibrium was established only after a week. Results are shown in Table 5. It is evident that zirconium and hafnium oxynitrates dissolve only slightly, or not at all, in slightly polar or nonpolar organic solvents. The compounds studied are unsoluble in acetophenone, dibutyl and benzyl ethers, chloroform, carbon tetrachloride, and dichloro ethane. The solubility of oxynitrates decreases with increasing chain length and

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branching of the saturated alcohols used as solvents. Under equal conditions, oxynitrates of zirconium are better soluble in organic solvents than those of hafnium. There are 4 figures, 5 tables, and 7 references: 4 Soviet and 3 non-Soviet. The three references to English-language publications read as follows: Ref. 4: B. Lister, L. McDonald, J. Chem. Soc., 1952, 4315; Ref. 5: R. Connick, W. McVey, J. Am. Chem. Soc., 71, 3182 (1949); Ref. 6: R. Connick, N. McVey, J. Am. Chem. Soc., 73, 1171 (1951).

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M. V. Lomonosova (Moscow Institute of Fine Chemical Technology imeni M. V. Lomonosov)

PRESENTED: April 25, 1961, by V. I. Spitsyn, Academician

SUBMITTED: April 22, 1961

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S/020/61/141/002/014/027
B103/B110

AUTHORS: Men'kov, A. A., Komissarova, L. N., Simanov, Yu. P., and Spitsyn, Vikt. I., Academician

TITLE: Scandium chalcogenides

PERIODICAL: Akademiya nauk SSSR. Doklady, v. 141, no. 2, 1961, 364-367

TEXT: High-purity Sc_2O_3 (of 99.9% purity), Sc_2S_3, and ScTe (the latter two synthesized from elements) were studied by x-ray diffraction. Compounds of the composition 1:1 were not found in the systems Sc - S and Sc - Se. PKA-57 (RKD-57) and PKU-86 (RKU-86) cameras with filtered CuK_α radiation were used for taking x-ray photographs. Results are given in Tables 1 - 3. All Sc_2O_3 lines are satisfactorily indicated in a cubic, body-centered Mn_2O_3 lattice with a = 9.835 ± 0.005 kX, Z = 16, which is somewhat more than the lattice constants given in publications. The density of Sc_2O_3 (g/cm^3) determined by x-ray diffraction is 3.84, the

Card 1/03

pycnometrically determined density is 3.75. As to their intensities, Sc_2S_3 lines may be clearly classified into two groups: (1) very strong ones, (2) weak ones. The former are indicated in a primitive cubic lattice with a_o = 2.591 kX which represents a subcell. The latter are due to a superstructure. In analogy with the structure of β-In_2S_3 (Ref. 12, see below), a tetragonal face-centered lattice, a = 10.37 ± 0.01 kX ($a = a_o \cdot 4$) and c = 31.11 ± 0.03 kX; c/a = 3, Z = 32, is assumed. The existence of lines which cannot be indicated is explained by an additional Sc_2S_3 superstructure, or by small impurities. The calculated packing density of such a tetragonal lattice was 2.96 g/cm^3, the pycnometrically determined one (in chloroform) 2.80 g/cm^3. All 25 lines of the ScTe photograph are well indicated in a hexagonal NiAs lattice with a = 4.112 ± 0.005 kX and c = 6.735 ± 0.005 kX, c/a = 1.634, Z = 2. The density of ScTe determined by x-ray diffraction is 5.75 g/cm^3, the pycnometrically determined one (in bromoform) is 5.65 g/cm^3. The results obtained are not in agreement with those of Ref. 13 (see below). The color of Sc chalcogenide changes regularly from white to black with increasing

Scandium chalcogenides

S/020/61/141/002/014/027
B103/B110

chalcogen polarizability. Sc_2O_3: white, Sc_2S_3: yellow, Sc_2Se_3: brown-violet, Sc_2Te_3: black, ScTe: black. The crystal lattices of these chalcogenides is of high symmetry. There are 4 tables and 13 references: 4 Soviet and 9 non-Soviet. The three references to English-language publications read as follows: H. E. Swanson, R. K. Fuyat, G. M. Ugrinik, National Bureau of Standards, Circular 539, 3, 1954; C. J. M. Rooymans, J. Inorg. and Nucl. Chem., 11, no. 1, 78 (1959); L. H. Brixner, J. Inorg. and Nucl. Chem., 15, No. 1/2, 199 (1960).

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova (Moscow State University imeni M. V. Lomonosov)

SUBMITTED: June 24, 1961

Card 3/3

KOMISSAROVA, L.N., kand. khim. nauk; SHEMANINA, V.N., red.; KUBKINA, V., tekhn. red.

[Hafnium] Gafnii; sbornik statei. Moskva, Izd-vo inostr. lit-ry, 1962. 364 p. (MIRA 15:4)
(Hafnium)

37519
S/020/62/144/001/014/020
B119/B144

AUTHORS: Men'kov, A. A., Komissarova, L. N., Karelin, V. V., Priselkov, Yu. A., Nesmeyanov, An. N., and Spitsyn, Vikt. I., Academician

TITLE: Investigation of high-purity metallic scandium

PERIODICAL: Akademiya nauk SSSR. Doklady, v. 144, no. 1, 1962, 122 - 125

TEXT: 99.5% pure Sc was produced by high-vacuum distillation of 97 - 97.5% Sc. The pure metal was studied metallographically and tested for its behavior to O_2, N_2 (in a device designed by R. D. Shapovalova and I. A. Vasil'yeva), and differently concentrated solutions of HCl, H_2SO_4, HNO_3, and NaOH at 25, 50, and 100°C. The results were compared with those obtained for 97% Sc. The polished, non-etched surface of 97% Sc reveals the grain boundaries in polarized and nonpolarized light. No second phase appears in spite of 0.9% oxygen content. With high-purity Sc, the grain boundaries are only visible in polarized light. 99.5% Sc starts reacting at 200°C with O_2, at more than 600°C with N_2 (formation of ScN). Dissolving

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SU

Card 1/2

40185

S/020/62/145/005/016/020
B106/B144

AUTHORS: Tsirel'nikov, V. I., Komissarova, L. N., and Spitsyn, Vikt. I., Academician

TITLE: Study of vapor density of hafnium tetrachloride at high temperatures

PERIODICAL: Akademiya nauk SSSR. Doklady, v. 145, no. 5, 1962, 1081-1084

TEXT: The vapor density of hafnium tetrachloride was determined at high temperatures with the aid of the radioisotope Hf^{181} following a method described by F. S. Dainton, H. M. Kimberley (Trans. Farad. Soc., 46, 912 (1950)) and V. V. Illarionov, S. A. Cherepanova (DAN, 133, 1086 (1960)). The tagged $HfCl_4$ contained 0.3% $ZrCl_4$ and <0.001% Fe, Ti, Si, Al. Isothermal and isobaric studies were carried out. The radiometric measurements were made whilst heating and cooling, and the resulting values showed good agreement (Fig. 3). The experimental setup consisted of two electric furnaces arranged at right angles. The vessels in the furnaces were joined by a capillary tube heated to 550°C; of such size that no noticeable pressure drop occurred. In the isothermal measurements, the

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S/020/62/145/005/016/020
B106/B144

Study of vapor density ...

first furnace was kept at constant temperatures between 400 and 1000°C and the temperatures in the second furnace were varied between 280 and 350°C, whereas in the isobaric tests, those temperatures were varied in the first and kept constant in the second furnace. The error of measurement was ± 2.5%. At high temperatures and low pressures, the hafnium tetrachloride vapor follows the ideal gas laws if it is assumed to be monomolecular. According to data of W. Fisher (Zg. anorg. u. allgem. Chem., 211, 321 (1933)), zirconium tetrachloride vapor behaves analogously. At a pressure increase above atmospheric pressure and a temperature decrease to near sublimation temperature, the vapor density of the real $HfCl_4$ vapor is as much as 10% lower than the ideal vapor density. This deviation cannot be explained by association, but by the nonideality of the vapor near condensation temperature. The data obtained are required for calculating technological processes to separate zirconium and hafnium tetrachlorides in the gaseous state. There are 3 figures and 2 tables. The most important English-language reference is: A. A. Palko, A. D. Ryon, D. W. Kuhn, J. Am. Chem. Soc., 62, 319 (1958).

Card 2/3

S/020/62/146/001/013/016
B101/B144

AUTHORS: Tsirel'nikov, V. I., Komissarova, L. N., and Spitsyn, Vikt. I., Academician

TITLE: Study of the thermal stability of the molecules of zirconium and hafnium tetrahalides on collision with a hot surface in vacuo

PERIODICAL: Akademiya nauk SSSR. Doklady, v. 146, no. 1, 1962, 122 - 124

TEXT: The decomposition coefficients of Zr and Hf tetrahalides excepting the fluorides were determined. The tetrahalides were heated in an ampoule with capillary outlets (Fig. 1) so that the effusion followed the Knudsen cosine law. The molybdenum target was heated to 1000 - 1500°C and the samples evaporated in vacuo at 10^{-6} mm Hg. On dissolving the target in HNO_3 the Zr or Hf was precipitated as metallic film, and weighed. Spectrum analysis showed a slight diffusion of Mo from the target into Zr or Hf. The amount of Zr or Hf precipitated and that of tetrahalide evaporated were used to calculate the decomposition coefficient K_d. At 1500°C this

Card 1/3

was 100% for ZrI_4, 90% for HfI_4, 68% for $ZrBr_4$, 61% for $HfBr_4$. There was almost no dissociation in the case of the tetrachlorides. K_d is proportional to the target temperature. The thermal stability of Zr and Hf tetrahalides corresponds to their formation enthalpies. There are 2 figures and 2 tables.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova (Moscow State University imeni M. V. Lomonosov)

SUBMITTED: March 26, 1962

Fig. 1. Apparatus for determining K_d of Zr and Hf tetrahalides.

Legend: (1) Apparatus, (2) extension of the ampoule, (3) heater, (4) molybdenum target, diameter 12-15 mm, thickness 0.5 mm, (5) molybdenum lead-in, (6) sight hole, (7) trap, (8) pyrometer, (9) liquid nitrogen, (10) ampoule.

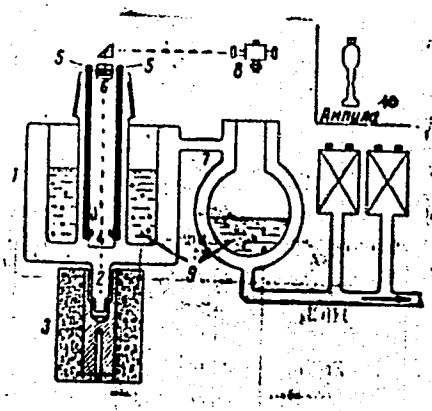

Fig. 1

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S/078/62/007/010/001/008
B144/B186

AUTHORS: Shatskiy, V. M., Kommissarova, L. N., Spitsyn, Vikt. I.

TITLE: Precipitation of scandium hydroxide and oxalate

PERIODICAL: Zhurnal neorganicheskoy khimii, v. 7, no. 10, 1962, 2294-2298

TEXT: 1) Effects of Sc concentration, nature and quantity of precipitant, and the influence of NH_4Cl on the precipitation degree of $Sc(OH)_3$ were studied in the concentration range of 1 - 60 g/l Sc_2O_3, with NH_4Cl additions of 50 - 150 g/l. The precipitants used were 10% and 25% solutions of NH_4OH and NaOH with no CO_2. Their content in the filtrate was 10 - 125 g/l. The pH was varied from 6.5 to 9.5. With NH_4OH, precipitation was 100% in concentration intervals from 1 to 30 mg/ml Sc_2O_3, and 98.7% in the concentration 60 mg/ml Sc_2O_3. The $Sc(OH)_3$ precipitation was slightly reduced (99.7%) with high NaOH excess. In both cases, the Sc_2O_3 content in the filtrate did not exceed 1 mg/l. This

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Precipitation of scandium...

holds equally for precipitation from nitric solutions containing 40 - 50% of rare-earth elements, 20% Th and 5% Sc (as calculated for oxides) and small amounts of Fe, Al, Mg, and Ca. 2) Degree of precipitation of Sc oxalate was studied as a function of relative concentrations of Sc_2O_3 and $H_2C_2O_4$. Precipitation from solutions containing 5 g/l Sc_2O_3 is 97%, and there is no influence of the precipitant within 100 - 300%. The precipitation degree exceeds 99.6% when the initial solution contains 50 - 100 g/l Sc_2O_3. In precipitation from 1 g/l Sc_2O_3 solutions, the precipitation rate decreases from 88.9 to 58.1% in dependence of the $H_2C_2O_4$ excess. If water (at 25°C) is used as a washing liquid the Sc content is 65 - 150 mg/l Sc_2O_3. Deviations from the results obtained by R. C. Vickery (J. Chem. Soc. (London), 3113 (1956)) are explained by inadequate radiometric analysis methods. There are 2 figures and 2 tables.

SUBMITTED: January 18, 1962

Card 2/2

ZELIKMAN, A.N., prof, doktor tekhn. nauk, red.; KOMISSAROVA, L.N., dots., kand. khim.nauk, red.; KRAPUKHIN, V.V., dots., kand. tekhn. nauk, red.; SEVRYUKOV, N.N., prof., doktor tekhn. nauk, red.; KAMAYEVA, O.M., red. izd-va; MIKHAYLOVA, V., tekhn. red.

[Separation of rare metals having similar properties]Razdelenie blizkikh po svoistvam redkikh metallov. Moskva, Metallurgizdat, 1962. 264 p. (MIRA 15:8)
(Nonferrous metals—Metallurgy)

S/153/62/005/004/001/006
E075/E436

AUTHORS: Korovin, S.S., Mironenko, A.P., Reznik, A.M., Komissarova, L.N.

TITLE: Extraction of hydrochloric acid and some elements with acetophenone

PERIODICAL: Izvestiya vysshikh uchebnykh zavedeniy, Khimiya i khimicheskaya tekhnologiya, v.5, no.4, 1962, 553-558

TEXT: The authors investigated the extraction of HCl with acetophenone (AF) and its solution in dichlorethane (4.28 mole/litre) from aqueous solutions. For pure acetophenone negligible amount of HCl is extracted from solutions containing less than 7 mole/litre HCl. The distribution coefficient increases rapidly above this concentration of HCl. It is postulated that monosolvate HCl.AF forms in the organic phase according to the equation

$$H^+ + Cl^- + AF \rightleftarrows HCl \cdot AF$$

The effective constant K for the complex formation was calculated to be 1×10^{-6}. HCl in the organic phase is ionized. Degree of dissociation α of HCl was calculated to be

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S/153/62/005/004/001/006
E075/E436

Extraction of hydrochloric acid ...

approximately 0.03, 0.48 and 0.88 for 0.28, 2.80 and 4.07 mole/litre HCl respectively using the formula $\alpha = \lambda\eta/60$ where λ is the electrical conductivity and η - viscosity of the solutions. For the extractions with the acetophenone-dichlorethane solution, distribution coefficients for HCl are small even at its very high concentrations. Dissociation of HCl does not occur in the mixed solvents. The latter was used for the extraction of Ca, Ga, Al, Zr, Hf and Fe^{3+} from aqueous solutions of varying acidity. The most extractable elements were Fe and Ga, their distribution coefficients being 34 and 44 respectively for the HCl concentration of 7 mole/litre. Zr and Hf begin to be extracted from the HCl solution of 8 mole/litre, but distribution coefficients are lower than for Ga and Fe. Coefficient of separation of Zr from Hf ($\beta = \alpha_{Zr}/\alpha_{Hf}$) increases with acidity and reaches the maximum value of 5 in 10.3 to 20.5 mole/litre HCl. It was found that the distribution coefficient for Zr decreases from 3.07 to 0.33 and the coefficient for Hf from 0.85 to 0.21, when the temperature of the solution (10.5 mole/litre HCl) increased from 20 to 60°C. There are 4 figures and 4 tables.

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KOMISSAROVA, L.N., kand. khim. nauk, red.; PLYUSHCHEV , V.Ye., doktor khim. nauk, red.; ALEKSEYEV, V.A., red.; KARPOV, I.I., tekhn. red.

[Metallurgy of rare earth metals]Metallurgiia redkozemel'nykh metallov; sbornik statei. Moskva, Izd-vo inostr. litry, 1962. 199 p. (MIRA 15:9)
(Rare earth metals--Metallurgy)

S/828/62/000/000/014/017
E071/E135

AUTHORS: Komissarova, L.N., Shatskiy, V.M., Zazubin, A.I., Savrukova, G.D., and Spitsyn, V.I., Academician.

TITLE: Separation of scandium from tungsten and poor polymetallic iron ores

SOURCE: Razdeleniye blizkikh po svoystvan redkikh metallov. Mezhvuz. konfer. po metodam razdel. blizkikh po svoyst. red. metallov. Moscow, Metallurgizdat, 1962, 155-167.

TEXT: As a result of experiments carried out with tungsten residues and slag, two methods of separation of scandium and production of a pure scandium oxide (above 99.99%) with an overall yield of 80-88% production, were developed. The first stage in both is the transfer of scandium into solution. The best results were obtained by treating the residues or slag with 98% sulphuric acid, using a solid to liquid ratio of 1:1, a temperature of 220 °C up to a nearly complete removal of SO_3 vapour (> 4 hours) and subsequent extraction with water. The solubility of $Sc(OH)_3$ in Na_2CO_3 solutions of various concentrations was studied at 0° and 25 °C. With increasing concentration of Na_2CO_3 the solubility

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of $Sc(OH)_3$ increases. The maximum solubility, 0.12 wt.% of $Sc(OH)_3$, is obtained at 20 wt.% of Na_2CO_3 and 25 °C. The solubility of $Sc(OH)_3$ in sodium hydroxide solutions in the range of concentration of 7-45 wt.% at 25 °C was determined. In the lower range of concentration of sodium hydroxide (up to 15 wt.%) the solubility of $Sc(OH)_3$ is insignificant ($\sim$ 0.03 mg Sc_2O_3 per mℓ of solution). The solubility was highest at 26 and 32.5 wt.% of NaOH, 1.28 and 1.5 mg of Sc_2O_3 per mℓ of solution. The above studies were used as a basis for the two proposed methods of separation. The carbonate method, proposed for the processing of tungsten residues, comprises: transfer into solution with concentrated sulphuric acid, sodium carbonate treatment, extraction of thiocyanides and precipitation of oxalates. The alkali-carbonate method, proposed for the separation of scandium from slags (from the production of pig iron) comprises: sulphuric acid solution, precipitation with sodium hydroxide, carbonate treatment, extraction of thiocyanides and precipitation of oxalates. As a result of the carbonate treatment 40-70% Sc_2O_3 concentrates are obtained. The main admixtures are thorium, rare earth elements,

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Separation of scandium from tungsten... S/828/62/000/000/014/017
E071/E135

zirconium, titanium, aluminium and berylium.
There are 2 figures and 7 tables.

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KOMISSAROVA, L.N.

1

S/153/62/005/002/003/004
E075/E435

AUTHORS: Korovin, S.S., Lebedeva, Ye.N., Reznik, A.M., Komissarova, L.N., Kuznetsova, G.P.

TITLE: Extraction of zirconium and hafnium with tributylphosphate

PERIODICAL: Izvestiya vysshikh uchebnykh zavedeniy. Khimiya i khimicheskaya tekhnologiya. v.5, no.2, 1962, 231-235

TEXT: The object of the work was to investigate distribution of Zr and Hf between nitric acid solutions and tributylphosphate (TBF). A 50% solution of TBF in o-xylene saturated with nitric acid was used as the extractant. Nitric acid concentration in the metal solutions was 6 mole/litre. Distributioh of Zr and Hf was studied for the solutions containing 2.4, 16.2, 50.0, 70.0, 95.8 and 100% Hf. It was established that the behaviour of Zr and Hf. is interconnected during the extraction but the influence of Zr on the extraction of Hf is more marked than the reverse influence. When a solution contains a predominant quantity of one of the metals, the extraction of the other metal is retarded. The maximum distribution coefficients (20.9) were obtained for the

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Extraction of zirconium and ...

solutions containing the smallest quantity of Hf (2.4% HfO_2). The coefficient decreases with the increasing concentration of Hf. When the concentration of the metals in the solution increases, the distribution coefficient increases and then decreases; thus, for Hf concentration of 50%, the coefficients are 5.8, 18.5 and 15.8 for the summed concentrations of the oxides in the solutions of 14.5, 73.6 and 92.1 g/litre respectively. It is concluded that the method can be used not only for the purification of Zr from Hf but also for the preparation of pure Hf. There are 3 figures and 1 table.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M.V.Lomonosova, Kafedra tekhnologii redkikh i rasseyannykh elementov (Moscow Institute of Fine Chemical Technology imeni M.V.Lomonosov, Department of Rare and Dispersed Elements Technology)

SUBMITTED: October 17, 1960

YURANOVA, L.I.; KOMISSAROVA, L.N.; PLYUSHCHEV, V.Ye.

Solubility and thermal stability of zirconium and hafnium oxynitrates hexahydrates. Zhur.neorg.khim. 7 no.5:1062-1067 My '62. (MIRA 15:7)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni Lomonosova.

(Zirconium nitrate) (Hafnium nitrate)

TSIREL'NIKOV, V.I.; KOMISSAROVA, L.N.; SPITSYN, Vikt.I.

Corroding effect of zirconium tetrachloride vapors on 1Kh18N9T steel and nickel at high temperatures. Atom. energ. 13 no.1: 51-53 Jl '62. (MIRA 15:7)

(Corrosion and anticorrosives)

MEN'KOV, A.A.; KOMISSAROVA, L.N.; KARELIN, V.V.; PRISELKOV, Yu.A.; NESMEYANOV, An.N.; SPITSYN, Vikt.I., akademik

Investigation of high-purity metallic scandium. Dokl.AN SSSR 144 no.1:122-125 My '62. (MIRA 15:5)

1. Moskovskiy gosudarstvennyy universitet im. M.V.Lomonosova. (Scandium)

KOMISSAROVA, L.N.; PUSHKINA, G.Ya.; SPITSYN, Vikt. I.

Preparation and some properties of scandium nitrates. Zhur. neorg. khim. 8 no.6:1384-1394 Je '63. (MIRA 16:6)

1. Moskovskiy gosudarstvennyy universitet imeni Lomonosova, kafedra neorganicheskoy khimii.
(Scandium nitrate)

S/032/63/029/001/014/022
B104/B186

AUTHORS: Granovskiy, Yu. V., Chernova, N. A., Adler, Yu. P., Nalimov, V. V., Komissarova, L. N., and Spitsyn, Vik. I.

TITLE: A mathematical model for the extractive separation of hafnium and zirconium by tributyl phosphate

PERIODICAL: Zavodskaya laboratoriya, v. 29, no. 1, 1963, 60-65

TEXT: Improvement of the conditions for separating zircon and hafnium from nitric acid solutions using tributyl phosphate is studied by the Box-Wilson method (G. E. Box, K. B. Wilson, J. Roy Stat. Soc. (B), 13, 1 (1951)). The following independent variables were selected: X_1 is the concentration of the metals for the sum of $Zr(Hf)O_2$ (g/l); X_2 is the concentration of the acid in the aqueous initial solution (gramm equivalent/liter); X_3 is the concentration of the tributyl phosphate in o-xylene (volume-%); X_4 is the phase ratio $V_o : V_B$. The optimization parameter is the separation factor y. Different series of experiments

show the appropriate programming matrices with the results. These are used to determine the direction in which the independent variables must be varied. For the separation factor the regression equation

$$\begin{aligned} y = {} & 13{,}3478 - 0{,}1496X_1 + 1{,}5036X_2 - \\ & - 0{,}6393X_3 + 0{,}2635X_4 + 0{,}1078X_1^2 - \\ & - 1{,}3422X_2^2 - 0{,}7798X_3^2 + 0{,}0200X_4^2 - \\ & - 0{,}0181X_1X_2 + 0{,}4756X_1X_3 + \\ & + 0{,}6432X_1X_4 - 0{,}1431X_2X_3 - \\ & - 0{,}0506X_2X_4 + 0{,}1931X_3X_4. \end{aligned}$$

is obtained, where $X_i = (\widetilde{X}_i - \widetilde{X}_{io})/\widetilde{X}_{iA}$, $\widetilde{X}_i$ is here the value of the natural variable, $\widetilde{X}_{io}$ and $\widetilde{X}_{iA}$ are the values of the reference point in the phase space and the variation interval. This equation describes the experimental results. By displacement along the coordinate axes X_i, separation factors (22.8 and 28.2) could be obtained which were larger than those hitherto known. Further, the model can be used to compensate

APPROVED FOR RELEASE: 06/13/2000 CIA-RDP86-00513R000824120003-1"

A mathematical model for the ...
S/032/63/029/001/014/022
B104/B186

uncontrolled changes of one or several variables by changing other variables arbitrarily. There are 1 figure and 4 tables.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet i Gosudarstvennyy nauchno-issledovatel'skiy i proyektnyy redkometallicheskoy promyshlennosti
(Moscow State University and State Design and Planning Scientific Research Institute of the Rare Metals Industry)

S/032/63/029/001/015/022
B104/B186

AUTHORS: Komissarova, L. N., Granovskiy, Yu. V., Prutkova, N. M., Adler, Yu. P., Nalimov, V. V., and Spitsyn, Vik. I.

TITLE: Determination of optimal extraction conditions for microquantities of hafnium using tributyl phosphate

PERIODICAL: Zavodskaya laboratoriya, v. 29, no. 1, 1963, 65-68

TEXT: Optimum conditions for extracting microquantities of hafnium from nitric acid solutions using tributyl phosphate are sought by means of the Box-Williams method (V. V. Nalimov, Uspekhi khimii, 29, 11, 1362 (1960), Zavodskaya laboratoriya, v. 29, no. 1, 1963, 60, G. E. Box, K. B. Wilson, J. Roy Stat. Soc. (B), 13, 1 (1951)). Parameters: X_1 is the concentration of the nitric acid in the aqueous initial solution (N); X_2 is the concentration of tributyl phosphate in o-xylene (volume-percent); X_3 is the phase ratio ($V_o : V_B$); X_4 is the extraction time (min). The optimization parameter is the hafnium distribution factor y. Working from an arbitrarily

Card 1/2

TSIREL'NIKOV, V.I.; KOMISSAROVA, L.N.; SPITSYN, Vikt. I., akademik

Thermal stability of zirconium and hafnium tetrahalide molecules striking a hot surface in vacuo. Dokl. AN SSSR 146 no.1:122-124 S '62. (MIRA 15:9)

1. Moskovskiy gosudarstvennyy universitet im. M.V. Lomonosova.
(Zirconium halides) (Hafnium halides)

KOMISSAROVA, L.N.; SAVEL'YEVA, M.V.; PLYUSHCHEV, V.Ye.

New zirconium and hafnium hydroxyacetates. Zhur.neorg.khim. 8 no.1: 56-62 Ja '63. (MIRA 16:5)

1. Moskovskiy gosudarstvennyy universitet imeni M.V.Lomonosova i Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni M.V.Lomonosova.
(Zirconium compounds) (Hafnium compounds) (Glycolic acid)

S/032/63/029/003/011/020
B112/B186

AUTHORS: Granovskiy, Yu. V., Nikishova, V. V., Adler, Yu. P., Nalimov, V. V., and Komissarova, L. N.

TITLE: Sifting experiments for investigating the extraction of zirconium from tributylphosphate

PERIODICAL: Zavodskaya laboratoriya, v. 29, no. 3, 1963, 321 - 326

TEXT: Those influences which predominatingly affect the process of extraction of zirconium from tributylphosphate are selected by the method of random balance. The following variables are codified: concentration of the metal (A), concentration of the acid (B), concentration of the reagent (C), volume of the restricted phase (D), extraction time (E), revolution velocity of the mixers during extraction (F), volume of the re-extragent (G), number of re-extractions (H), re-extraction time (I), revolution velocity of the mixers during re-extraction (J), time of phase separation after extraction (K), time of phase separation after re-extraction (L). The results of the experiments are represented in the dispersion diagram (Fig. 1). The selection of the predominating effects A, B, C, AB, BC, and CD was obtained under conditions at which 78 effects (12 linear and 66 pair

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Sifting experiments for...

interactions) could be of importance. There are 6 figures and 5 tables.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet i Gosudarstvennyy nauchno-issledovatel'skiy i proyektnyy institut redko-metallicheskoy promyshlennosti (Moscow State University and State Scientific Research and Planning Institute of Rare-Metal Industry)

S/032/63/029/003/012/020
B101/B186

AUTHORS: Komissarova, L. N., Granovskiy, Yu. V., Prutkova, N. M., Adler, Yu. P., and Nalimov, V. V.

TITLE: Application of mathematical experimental programming methods to studying the extraction of zirconium

PERIODICAL: Zavodskaya laboratoriya, v. 29, no. 3, 1963, 327 - 330

TEXT: For extracting zirconium by means of tributylphosphate (TBP) three possible reaction equations are written down: $Zr^{4+} + 4NO_3^- + TBP \rightleftharpoons Zr(NO_3)_4 \cdot TBP$; $Zr^{4+} + 4NO_3^- + 2TBP \rightleftharpoons Zr(NO_3)_4 \cdot 2TBP$; $Zr^{4+} + 2H^+ + 4NO_3^- + 2TBP \rightleftharpoons Zr(NO_3)_4 \cdot 2TBP + H_2O$. The equations for the apparent extraction constants $\tilde{K}_e$ are linearized to: $\log D = \log \tilde{K}_{e_1} + 4 \log X_H + \log T$; $\log D = \log \tilde{K}_{e_2} + 4 \log X_H + 2 \log T$; $\log D = \log \tilde{K}_{e_3} + 6 \log X_H + 2 \log T$. Here X_H is the equilibrium concentration of the hydrogen ions, T is the concentration of

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the free TBP in the organic phase, D the distribution factor. The following independent variables were chosen for programming: $X_1 = \log_2 X_H - 1.5$ and $X_2 = 2(\log_2 T + 2.5)$. The dependent variable is $y = \log_2 D$. The regression equation $y = -4.2230 + 3.609236X_1 + 0.7768862X_2 + 0.7814312X_1^2 + 0.5988127X_2^2 + 0.000725X_1X_2$. The extraction was performed using TBP diluted with xylene. The distribution was examined with $1 \cdot 10^{-5}$ mole/l Zr^{95}. The value of X_H was varied from 1.053 to 7.50 and that of T from 0.108 to 0.250. The center of the experiment was close to $X_H = 2.83$, $T = 0.177$.

Results: None of the three reaction equations describes the extraction process correctly. The data obtained from the regression equation do not agree with the experimental ones. Sidereactions, as e.g. the formation of different solvates and complexes (such as the complex $H_{n-2}ZrO(NO_3)_n$) are likely to occur. Nevertheless the regression equation can be used to estimate D. Here the error is four times the experimental error. There are 2 tables.

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S/032/63/029/003/012/020
B101/B186

Application of mathematical experimental...

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University); Gosudarstvennyy nauchno-issledovatel'skiy i proyektnyy institut redkometallicheskoy promyshlennosti (State Design and Planning Scientific Research Institute of the Rare Metals Industry)

Card 3/3

GRANOVSKIY, Yu.V.; ADLER, Yu.P.; NALIMOV, V.V.; KOMISSAROVA, L.N.

Screening experiments in the study of separation of zirconium and hafnium by extraction with tributyl phosphate. Zav. lab. 29 no.10:1220 '63. (MIRA 16:12)

1. Moskovskiy gosudarstvennyy universitet i Gosudarstvennyy nauchno-issledovatel'skiy i proyektnyy institut redkometallicheskoy promyshlennosti.

KOMISSAROVA, L.N.; POKROVSKIY, B.I.

Thermal stability of ScF_3 and its reaction with MgF_2. Dokl.AN SSSR 149 no.3:599-601 Mr '63. (MIRA 16:4)

1. Moskovskiy gosudarstvennyy universitet im. M.V.Lomonosova. Predstavleno akademikom V.I.Spitsynym.
(Scandium fluoride) (Magnesium fluoride)

L 10173-63 FCS(f)/EWP(q)/EWT(m)/

BDS--AFFTC/ASD--JD/JG

ACCESSION NR: AP3001405 S/0020/63/150/004/0816/0819

61
60

AUTHOR: Komissarova, L. N.; Spitsyn, Vikt. I. (Academician); Wang, Ken-shih

TITLE: Lanthanum and neodymium hafnates

SOURCE: AN SSSR. Doklady, v. 150, no. 4, 1963, 816-819

TOPIC TAGS: lanthanum hafnate, neodymium hafnate, lanthanum oxide, neodymium oxide, HfO sub 2, phase diagram, semiconductor, refractories, chemical resistance, chlorinating agent, hydrofluoric acid

ABSTRACT: The synthesis of lanthanum and neodymium hafnates and certain of their physical and chemical properties have been studied. To investigate the conditions of the reaction of HfO sub 2 with lanthanum oxide or neodymium oxide, the required mixtures were prepared 1) by mechanical mixing of the initial oxides and 2) by coprecipitation of the hydroxides. Thermal and x-ray analysis and, in some cases, chemical phase analysis and electrical conductivity measurements were conducted. It was found that a new crystalline phase with a pyrochlore structure is formed -- in case (2) at low temperatures, probably in the course of dehydration,

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OTHER: 004

KOMISSAROVA, L.N.; KRASNOYARSKAYA, A.A.; GULIA, V.G.

Scandium thiocyanates. Zhur. neorg. khim. 9 no.2:477-478 F'64.
(MIRA 17:2)

1. Moskovskiy gosudarstvennyy universitet imeni Lomonosova, kafedra neorganicheskoy khimii.

KOMISSAROVA, L.N.; VAN GEN'-SHI [Wang Ken-shih]; SPITSYN, Vikt.I.; SIMANOV, Yu.P. [deceased]

System La_2O_3 - HfO_2 . Zhur. neorg. khim. 9 no.3:693-697 Mr '64. (MIRA 17:3)

1. Moskovskiy gosudarstvennyy universitet im. Lomonosova, kafedra neorganicheskoy khimii.

L 11399-65 EWT(m)/EPF(c)/EPR/EWP(t)/EWP(b) Pr-4/Ps-4 JD/JW/JG

ACCESSION NR: AP4046447 S/0078/64/009/010/2277/2279

AUTHOR: Komissarova, L. N.; Pokrovskiy, B. I.

TITLE: Thermal reduction of scandium fluoride by magnesium

SOURCE: Zhurnal neorganicheskoy khimii, v. 9, no. 10, 1964, 2277-2279

TOPIC TAGS: scandium, scandium fluoride, scandium fluoride reduction, magnesium scandium system, magnesium scandium alloy

ABSTRACT: The reduction of scandium fluoride (ScF_3) with metallic magnesium and changes occurring in the Mg-Sc system have been studied in an attempt to determine the feasibility of alloying magnesium with scandium by the addition of scandium fluoride. It was found that the [illegible] scandium in magnesium at [illegible] temperatures [illegible]. The solubility of scandium in magnesium [illegible] 2—2½%. The magnesium-scandium [illegible] is of a peritectic [illegible] (Fig. 1 of the Enclosure). The [illegible] scandium fluoride [illegible] magnesium was found to begin [illegible] immediately after